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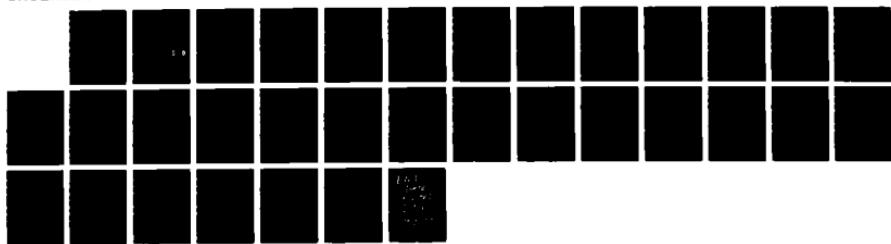
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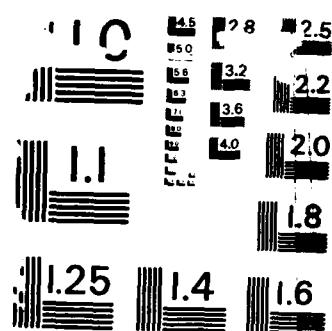
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An Electrochemical and Raman Spectroelectrochemical Investigation
of Underpotentially Deposited Silver on a Gold Substrate .

By

Stanley Pons, J. Li, J. Liang

Prepared for Publication in
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University of Utah
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AN ELECTROCHEMICAL AND RAMAN SPECTROELECTROCHEMICAL
INVESTIGATION OF UNDERPOTENTIALLY DEPOSITED SILVER ON
A GOLD SUBSTRATE

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ABSTRACT

Electrochemical and spectroelectrochemical data indicate that underpotentially deposited silver adatoms on gold substrates are photolyzed to form silver metal clusters. The surface gold-gold stretch and gold-silver stretch bands are observed, as well as the surface chloride complex stretch. The chloride complex interacts with ions in the solution phase giving rise to shifts in the metal-chloride stretching vibrational frequency as a function of the applied electrode potential. However, the metal-metal bands are more dependent on the electric field at the electrode surface.

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INTRODUCTION

The study of the interaction of adsorbates and surface complexes with the electric field at the electrode surface and with the solvent/ electrolyte in the electrical double layer is central to the understanding of the structure and character of the electrode solution interface. In situ spectroelectrochemical techniques coupled to conventional electrochemical methods provide much information about the structure and energetics of this interface. Surface infrared and surface enhanced Raman studies are now well established techniques in those investigations. Many workers now feel that the nature of the SERS phenomenon does not cloud the chemical interpretation of the spectra, at least to a first approximation; DSERS (diminished surface enhanced Raman scattering) and SUERS (surface unenhanced Raman scattering) apparently yield the same results (in terms of frequency and relative intensities) as does SERS; the major differences being simply the decreased intensities these latter techniques. It is also important to now compare directly the results of Raman and surface infrared spectroscopy, since the two methods are governed by different sets of selection rules, and must therefore provide complementary information of the vibrational structure of the interfacial region.

Studies of surface interactions are facilitated when the surface coverage of the adsorbate is carefully controlled. One way to accomplish this is the relatively simple technique of underpotential deposition of a metal on the electrode surface. On commonly studied system is silver underpotential deposition on gold substrates. In this work, we investigate the effects of surface pretreatment on the adsorption of, and the role of the photolytic excitation frequency on the surface chemistry of silver adatoms on gold.

EXPERIMENTAL

Silver nitrate (certified reagent) and perchloric acid (70% reagent) were obtained from Fisher Scientific and were used as received. Potassium chloride (AR, Mallinckrodt) and acetonitrile (HPLC Distilled in Glass, Burdick and Jackson) were used as received. Lithium perchlorate (anhydrous reagent, Aldrich Chemical) was dried in vacuo at 150°C for 24 hours, and stored in vacuo prior to use. Water was triply distilled.

The reference electrode for the experiments in acetonitrile was a silver/ silver ion type (0.01M silver nitrate and 0.10M tetrabutylammonium fluoroborate in acetonitrile). It was isolated from the working solution by a compartment which was terminated by a Luggin capillary tip which was located close to the working electrode. The reference electrode for the measurements in aqueous solution was a standard calomel (SCE) type which was similarly isolated.

A 3.5mm diameter gold disk electrode was used for all electrochemical measurements, and a 10mm gold disk was used for all spectroelectrochemical experiments. The electrodes were mounted in a Teflon housing. The electrochemical cell was a three compartment type designed such that all three electrodes were isolated. The spectroelectrochemical cell was of a type standard for reflection SERS or reflection infrared measurements. The position of the electrode with respect to the optical window or the Luggin capillary could be adjusted. The cells were cleaned in boiling sulfuric/ nitric acid, rinsed, and steamed with boiling triply distilled water before use.

The gold electrodes were mechanically polished successively with 1.0-, 0.3-, and 0.05 μm alumina on a balsa wood base to a mirror finish. They were then cleaned in an ultrasonic bath containing triply distilled water. Prior to

some measurements, the electrodes were then activated, by cycling them in an aqueous solution of 1M potassium perchlorate from -1.0 to +1.1V vs. SCE. The electrodes were electrochemically roughened in some experiments by the method of Gao et al (1) by voltammetry for 30 cycles from -0.1 to +1.1 V vs. SCE in a solution of 0.1M potassium chloride at 100 mV s⁻¹ potential sweep rate. After this roughening treatment, the surface had a matte, nonuniform brownish appearance. The electrodes were finally rinsed with triply distilled water (and dried in a vacuum oven prior to use in the anhydrous acetonitrile solutions).

When making spectroelectrochemical measurements, the cell was fitted with a quartz window. The cell was mounted on a three dimensional and two rotational micromanipulator stages. The laser incidence angle could be varied, and the laser spot could be scanned across the electrode surface. The incidence angle was maintained at 57° so that the beam reflected from the surfaces of the cell window and the electrode surface could be excluded from the spectrometer collection optics.

The Raman system for the surface enhanced scattering experiments consisted of a Spex 1401 double monochromator. The instrument had an 1800 lines mm⁻¹: holographic gratings. The spectrometer and detector were interfaced to a Nicolet 1180 computer system for spectrometer control, data acquisition, analysis, and graphic plotting. Laser excitation was provided by a Coherent Radiation Innova 90 krypton ion laser, and a Spectra Physics 165 argon ion laser. In this work, the excitation wavelengths used were 647.1, 568.2, 514.5, and 488.0 nm. Laser power was typically between 60 and 140 mW. The spectral bandpasses were 4cm⁻¹ for the 647.1 and 568.2 nm excitations and 5cm⁻¹ for the 514.5 and 488.0 nm excitations. Detection was by a Ga-As photocathode C31034 photomultiplier tube which was cooled by a thermoelectric refrigerator (Products

for Research, Inc.). Scattering data was acquired at 2cm^{-1} intervals with a 0.5s integration interval. Spectra were finally obtained by recovery from the computer. The spectrometer was calibrated with carbon tetrachloride using the 647.1 and 568.2 nm excitation wavelengths. There were less than 1cm^{-1} errors at each excitation for the characteristic bands for carbon tetrachloride.

RESULTS AND DISCUSSION

The activated smooth and roughened gold electrode surface.

The electrochemical behavior of the roughened gold surface is very similar to that of the activated, smoothly polished gold surface, although its appearance is different in terms of color and reflectivity. Comparisons of the cyclic voltammetry and differential capacity curves measured in aqueous 0.1M potassium chloride and 1M perchloric acid, and 1M lithium perchlorate acetonitrile solution show no significant differences for each electrode. The roughness factors are also apparently similar from estimates made from the differential capacitance data. As we shall see, however, the SERS intensity from on the roughened gold surface is 3-5 times larger than on the polished mirror surface.

Silver underpotential deposition on gold from aqueous solution

The cyclic voltammetry for silver underpotential deposition on a polished gold electrode from a 0.01M silver nitrate solution in 1M perchloric acid (Figure 1) is similar to those reported by Schmidt and Stuki for silver underpotential

deposition from a 2.6×10^{-5} M silver ion solution in 0.5 M sulfuric acid [2] and by Lorenz et al [3] for silver underpotential deposition from 3.2×10^{-4} M silver nitrate in 1 M perchloric acid. Lorenz reported two deposition peaks at 0.83 and 1.2V vs. the SCE, with a bulk deposition potential at 0.578V. In this work, the underpotential deposition peak was observed at 0.67V, and the stripping peak at 0.69V. The underpotential deposition current and the stripping current are proportional to the voltammetric potential sweep rate. The bulk silver deposition potential was observed to begin at 0.55V. On an anodic sweep, a wave is observed starting at +0.95V due to oxidation of the gold surface. For comparison, all of the experiments were run in the absence of silver ion. No peaks were observed over the same range of potentials. The deposition isotherm (Figure 2) shows a dip at around 0.7V. The charge under the underpotential deposition peaks corresponds to $235 \mu\text{C}/\text{cm}^{-2}$ at 100% monolayer coverage. Results for the roughened gold surface were virtually identical.

Results of the Raman experiments in the aqueous system

Roughened gold electrodes with underpotentially deposited silver

Red (647.1 nm) excitation

The Raman scattering was measured on the gold electrode system in the absence of and in the presence of silver ion (Figure 3). In the absence of silver ion, bands were observed at 165 and 245cm^{-1} . When silver adatoms had been underpotentially deposited onto the gold electrode, an additional band was observed at 90cm^{-1} . The band could be resolved from the background scattering when the electrode potentials were made more negative than about 0.9V. The band, once resolved, has a virtually constant magnitude as the potential is made more

negative. The intensity of and the frequency of the band at 165cm^{-1} was independent of the electrode potential. This band is also observed on the dry activated smooth or roughened gold electrode measured ex situ to the cell. The band at 245cm^{-1} is dependent on the electrode potential (Figure 4).

Yellow (568.2 nm) excitation

When in potential regions where silver deposition occurs, a band at 128cm^{-1} is observed in solutions containing silver ion (Figure 5). This band cannot be detected if silver ion is absent. Compared to the result with the 647.1nm excitation, the intensity of the Au-Cl stretch band is very small. A band at 233cm^{-1} can also be detected with this excitation at the electrode in both solutions containing and not containing silver. In addition, this band is present at the dry roughened gold surface, but not at the dry polished gold surface.

Green (514.5nm) and blue (488nm) excitations

A very broad weak band at about 185cm^{-1} , which is likely scattered light from the window, is observed with the 514.4nm excitation on the electrodes. No bands at all are observed from the electrodes with 488nm excitation.

Raman results for silver underpotentially deposited on the activated Smooth gold electrodes

The results for the visibly smooth gold electrode were essentially identical to

those for the roughened surface, except that the scattering intensities were at least an order of magnitude smaller.

Discussion of the aqueous system.

The spectroelectrochemically determined assignments for the 90cm^{-1} band has not previously been made. An estimate of the Au-Ag stretch can be estimated from the value of the spectroelectrochemically determined Pt-H stretching frequency, which has been observed experimentally by the SNIFTIRS method (4) and by others ((5), at 700K) at 950cm^{-1} . The gas phase ground state vibrational frequencies for Pt-H and Au-H are similar to each other due to the similarity in the large mass of the two systems:

$$\nu(^2\Delta)_{5/2} \text{ Pt-H} = 2294.7\text{cm}^{-1} \quad \nu(^1\Sigma^+) \text{ Au-H} = 2305.1\text{cm}^{-1}$$

We assume that the electrode hydrogen atom adsorption stretch frequency on gold would then be similar (this has not been observed experimentally) to that of platinum-hydrogen. Under this assumption, we can make a simple mass-frequency assumption:

$$\frac{\nu \text{ Au-Ag}}{\nu \text{ Au-H}} = \left(\frac{M(\text{H})}{M(\text{Ag})} \right)^{0.5}$$

This give a value for the gold-silver stretch frequency of 91cm^{-1} , in excellent agreement with the value that we have observed in this work.

Matrix isolated silver atoms have been investigated (6). The Ag-Ag stretching frequency for the dimer has been determined in those studies to be

194cm⁻¹. We have not been able to observe this band in this work, presumably because it is most likely buried beneath the strong Au-Au 165cm⁻¹ band. Bulk Ag-Ag phase would be very close to the Au-Au band; gas phase metal stretch frequencies for gold and silver are 200 and 198cm⁻¹ respectively.

We have previously observed (7) that a 145cm⁻¹ band is due to the surface atom Au-Au stretch. In that work the system was investigated using the SNIFTIRS infrared spectroelectrochemical method. The observed potential dependency of that band in both experiments is similar. The two experiments differed in the respect that the infrared work was performed in non-aqueous conditions in a potential region some 2V more negative. In the previous work, it was observed that the intensity of the Au-Au band decreased in magnitude as lithium was deposited on the surface of the gold. Silver deposition on gold in the Raman experiment did not show such a marked decrease in intensity: this effect has been observed many times in surface Raman experiments. We conclude that the band at 165cm⁻¹ reported in this work is of the same origin.

The Au-Cl stretch band was reported by Gao et al (1) to be centered 265cm⁻¹. The SERS electrodes in this work were roughened in KCl solution, and then returned to perchlorate solutions for the underpotential deposition solutions. We have undoubtedly transferred the gold-chloride complex to the Raman cell in this operation. The interaction of specifically adsorbed hydrogen cation with the surface chloride would be expected to decrease the vibrational frequency of the surface complex by decreasing the local electron density of the Au-Cl bond. The 245cm⁻¹ band observed in this work is also seen in chloride solutions containing no perchlorate. The roughened SERS gold electrode in potassium chloride solution showed a band at 250cm⁻¹; we assign the 245cm⁻¹ band to be the Au-Cl surface complex stretch mode.

The intensity of the 245cm^{-1} band was observed to decrease as underpotential deposition of silver was in progress. This observation is in agreement with the data of Guy et al (8) who observed a decrease in the Raman intensity as Pb was underpotentially deposited on silver, and Kester (9) who observed a similar result when depositing thallium on silver.

The bands observed with red excitation, eg 165 and 245cm^{-1} cannot be clearly detected with excitation radiation of shorter wavelengths due to the absorption of the excitation radiation by the gold electrode (10). Reflectance data show that the onset of absorbance by gold is at 2.1eV (590nm). SERS data (11) show that the Raman scattering intensity from pure gold under 514.5 and 488nm excitations are very small. It is therefore reasonable to assume that we could not detect the surface complexes easily with the short wavelength excitations.

The band at 128cm^{-1} results most likely from the photolysis of silver adatoms on the gold surface. Takamura (11) has previously reported degradation of the electroreflectance signal for deposits of silver on gold. Silver atom aggregation initiated by photolysis has been reported in other experiments (6,12). It is possible that silver adatoms on the gold surface aggregate under the yellow light illumination to Ag_n , with $n > 2$.

Silver underpotential deposition on gold from acetonitrile solutions

The cyclic voltammetry for a gold electrode in 0.01M silver nitrate and 1M lithium perchlorate in acetonitrile solution shows three cathodic peaks (0.59, 0.42, and 0.22V vs the silver/silver ion reference), and on the reverse sweep, four anodic peaks (0.25, 0.42, 0.60, and 0.70V, Figure 6). The peak currents of all of these peaks are proportional to the sweep rate indicating adsorption/desorption phenomena. Kolb et al [13] have reported the underpotential deposition of silver adatoms on gold, but did not publish the voltammetry or the adsorption isotherms. We observe that the isotherm for silver underpotential deposition on gold from acetonitrile is approximately linear with the electrode potential (Figure 7). The charge at full coverage is $200\mu\text{C cm}^{-2}$. The underpotential deposition characteristics of the roughened gold electrode are virtually identical to the activated smooth gold electrode.

Results of the Raman experiments in the acetonitrile system

The surface enhanced Raman scattering spectra were recorded for the activated smooth and roughened gold electrodes as a function of laser excitation frequency and electrode potential. The spectra were again recorded in the presence of and in the absence of silver. The results for both electrodes were the same as those in the aqueous solutions (*vide supra*), except that the 245cm^{-1} band (Au-Cl) on the roughened electrode was much weaker and was not significantly dependent on the magnitude of the electrode potential. The silver cluster band (128cm^{-1}) is resolved from the Au-Au stretch band (165cm^{-1}). Again, the 165cm^{-1} band is most likely buried in the tail of the 128 and 135cm^{-1} bands.

Discussion of the acetonitrile system

The results imply that the chemistry and energetics of the surface adatoms is essentially the same in both solvent systems. The interactions of ions and solvent molecules in the electrical double layer with the adatoms are thus probably small, and the shift in frequency of the metal Au-Ag stretch is most likely due to a field effect. The Au-Cl stretch, however, is not observed to shift with potential in the acetonitrile solvent as it does in the aqueous system. This is the expected result if the shift were due to a chemical effect involving the activity of hydrogen in the electrical double layer. In fact, the double layer in this solvent is known to be diffuse (14), so that the activity of the perchlorate at the plane of the chloride complex will be smaller than it is in the aqueous solution.

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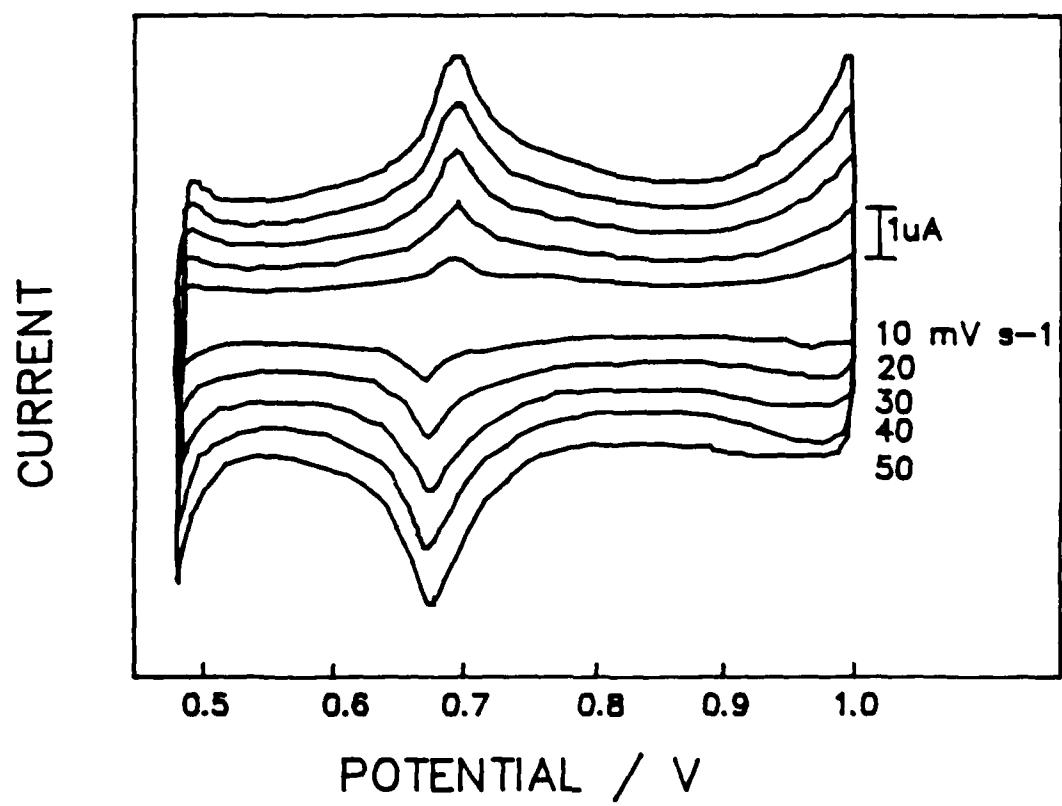
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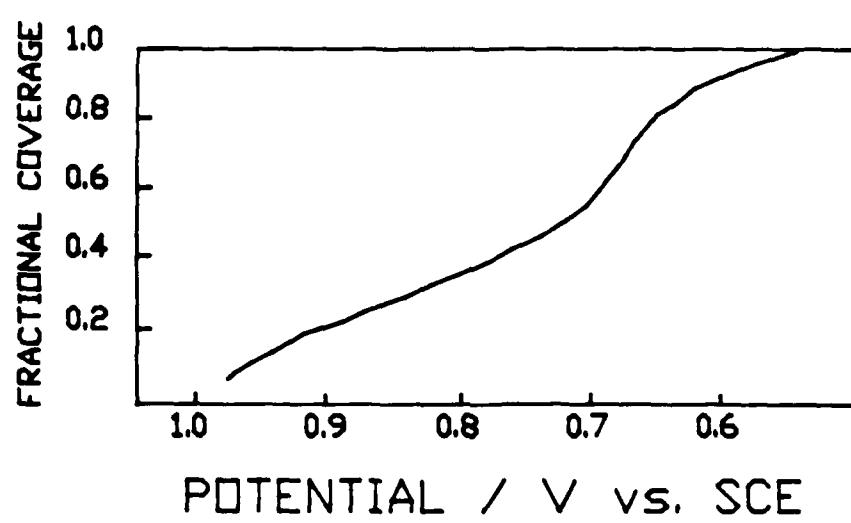
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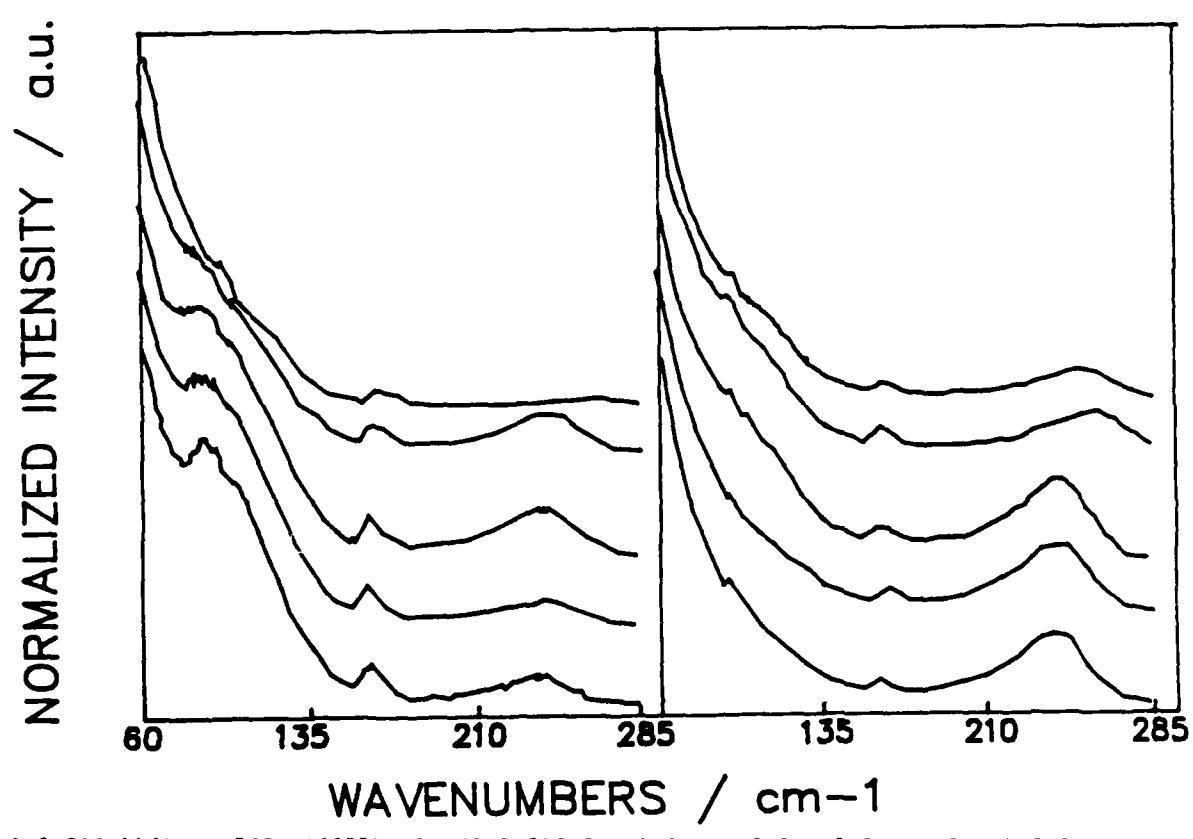
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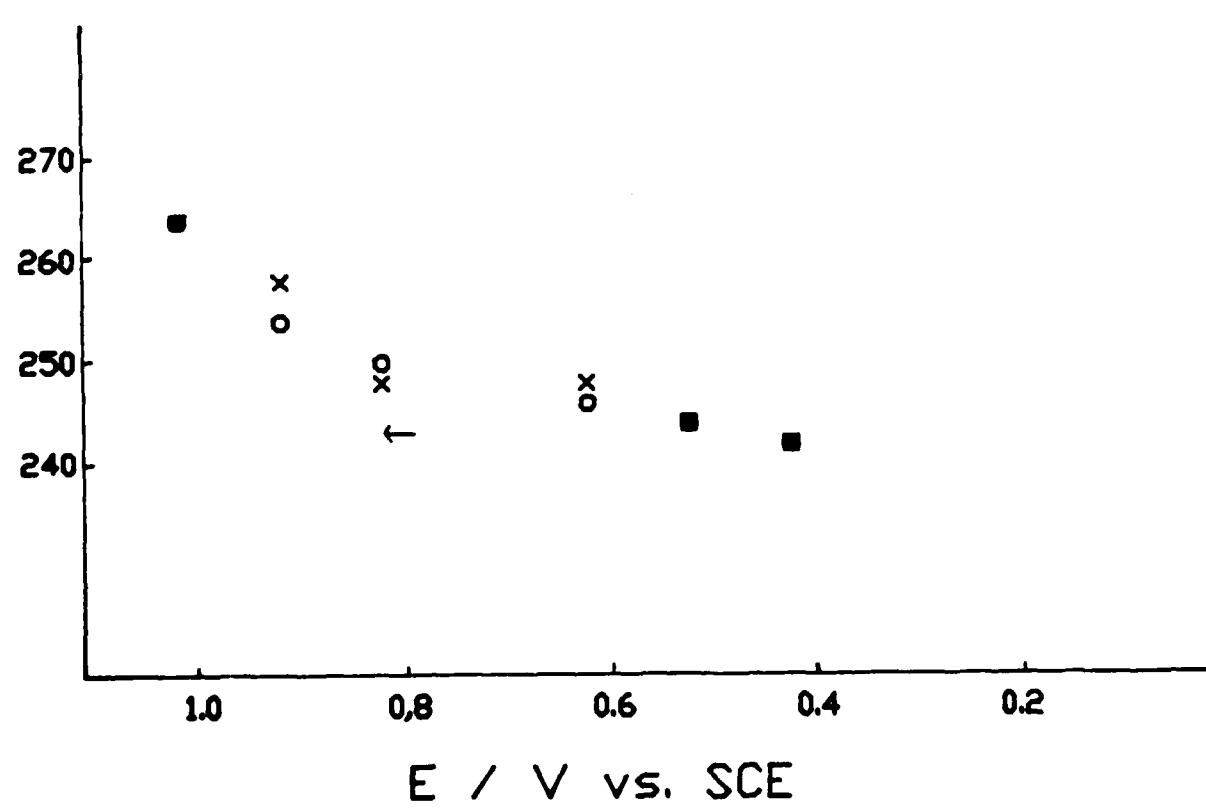
FIGURE LEGENDS

1. Voltammetric response for the aqueous 10^{-2}M silver nitrate, 1M perchloric acid solution at a gold electrode. The electrode was pretreated by cycling from +1.7 to -0.5V for 5 minutes at 100mV s^{-1} in the absence of silver ion.
2. Isotherm for silver under potential deposition on a roughened polycrystalline gold electrode in 10^{-2}M aqueous silver nitrate in 1M lithium perchlorate solution. The data is corrected for double layer charging. The maximum capacitance is $235\mu\text{C cm}^{-2}$ at a surface coverage of unity.2.
3. SERS spectra for the gold electrode in 1M aqueous perchloric acid solution (left) in the presence of 10^{-2}M silver ion, and (right) in the absence of silver ion. The laser excitation frequency was 647.1nm , and the power was 80mW . Other experimental conditions were identical in the two experiments.
4. (a) Au-Cl frequency shift with electrode potential at a roughened gold electrode in 1M aqueous perchloric acid solution. The circles indicate the result when 10^{-2}M silver nitrate is present, and the crosses are the result in the absence of silver ion. The arrow shows the result at the rest potential in 1M potassium chloride solution. (b) Au-Cl band intensity variation with electrode potential for the same system as above.
5. SERS spectra for the gold electrode in 1M aqueous perchloric acid solution in the presence of 10^{-2}M silver ion. The laser excitation frequency was 568.2nm , and the power was 80mW .
6. Voltammetric response for the acetonitrile 10^{-2}M silver nitrate, 1M lithium perchlorate solution at a gold electrode.
7. Isotherm for silver under potential deposition on a gold electrode in 10^{-2}M silver nitrate in 1M lithium perchlorate/ acetonitrile solution. The data is corrected for double layer charging. The capacitance is $200\mu\text{C cm}^{-2}$ at a surface coverage of unity.

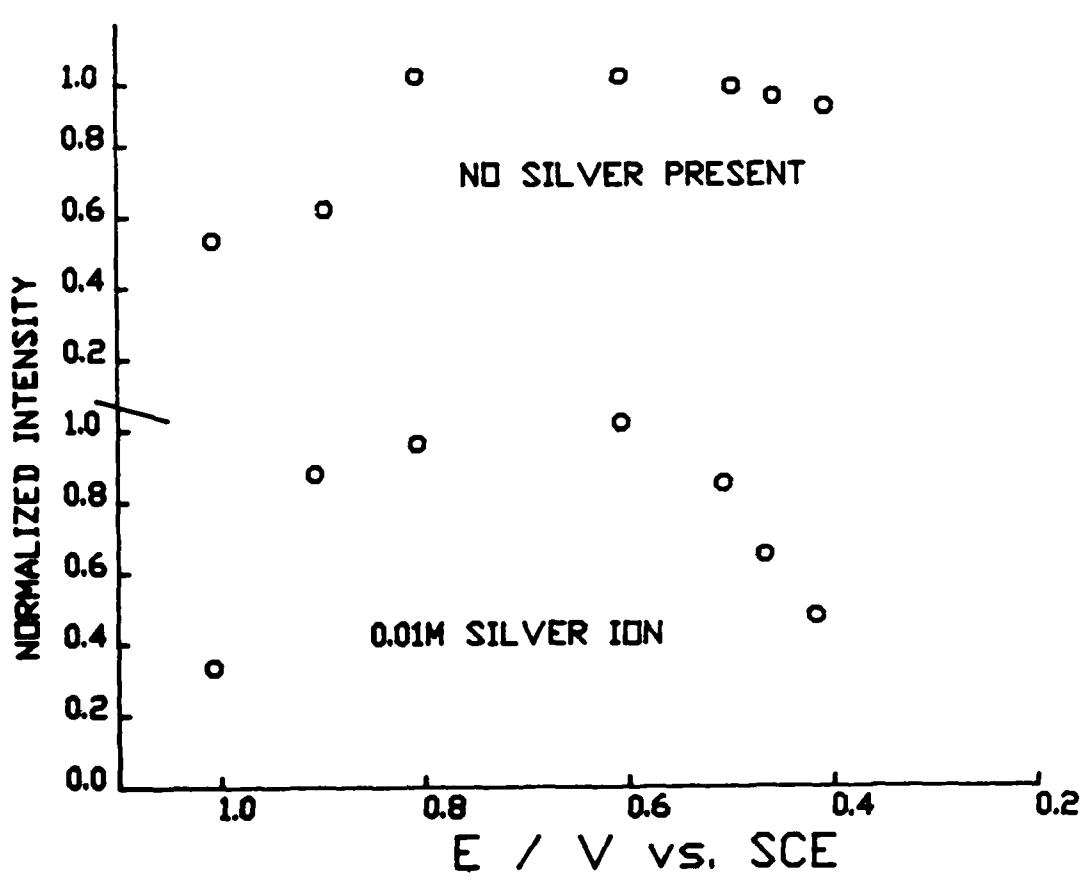


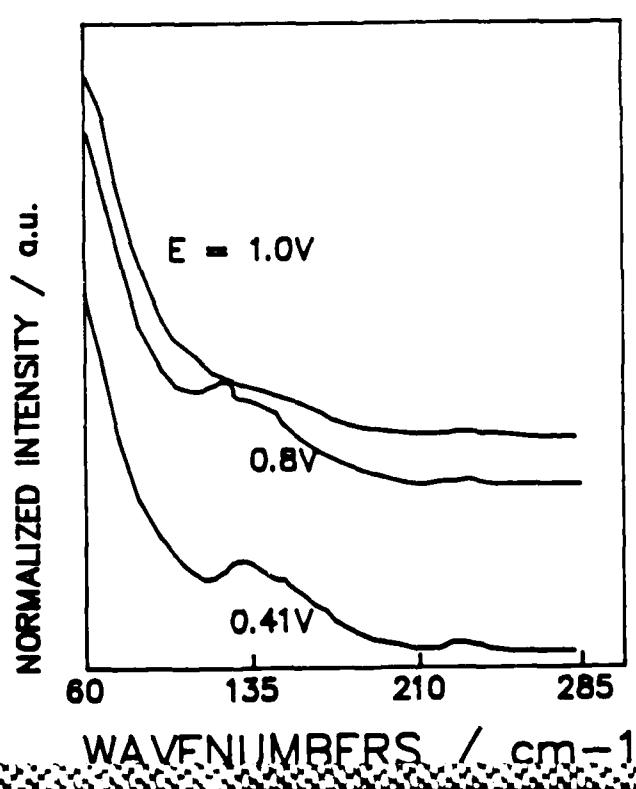


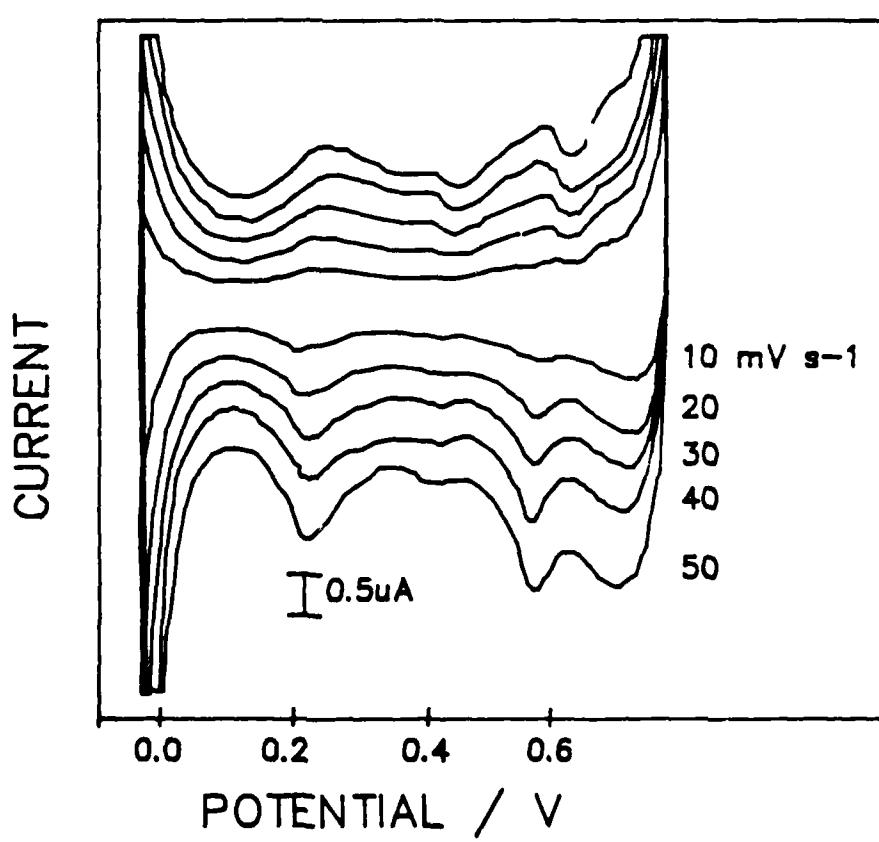


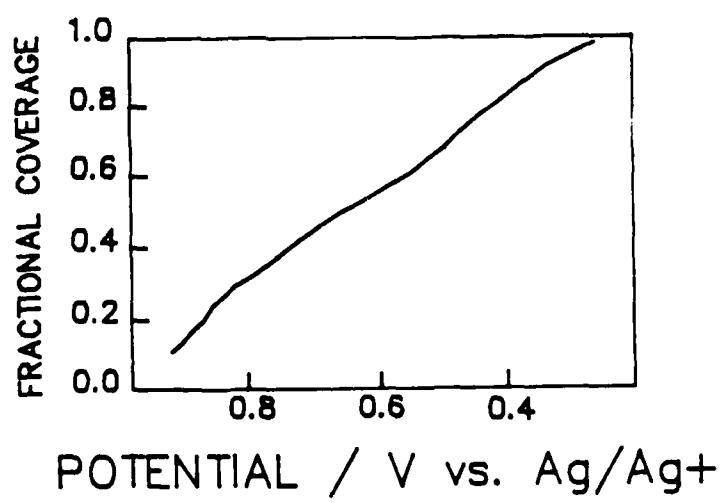


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